

# (12) UK Patent Application (19) GB (11) 2 324 615 (13) A

(43) Date of A Publication 28.10.1998

(21) Application No 9805452.1

(22) Date of Filing 16.03.1998

(30) Priority Data

(31) 9705446

(32) 15.03.1997

(33) GB

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(51) INT CL<sup>6</sup>

**B41N 1/24 , D21H 17/34 17/37**

(52) UK CL (Edition P )

**G2C CHX CH6D1**

**D1R RDH R162 R306 R309 R318 R641 R642 R645 R647**

(56) Documents Cited

**EP 0451269 A1**

**WPI Abstract Accession Number 95-118445/199516 &  
JP7040676**

(58) Field of Search

**UK CL (Edition P ) D1R RDH , G2C CHX**

**INT CL<sup>6</sup> B41N 1/24 , D21H 17/34 17/37**

(54) Abstract Title

**Paper**

(57) A paper comprises cellulose fibres and optionally synthetic polymer fibres therein the fibres of the paper are bonded together by a cured resin admixture comprising 0.05 gsm to 1 gsm of dry strength increasing additive, 0.1 gsm to 0.5 gsm of a cationic wet strength increasing agent, and 0.5 gsm to 3 gsm of an acrylate polymer, the admixture having been added and cured subsequent to the paper forming step in a single operation. The paper may be for use in a thermosensitive stencil paper.

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**PAPER**

The present invention relates to paper intended particularly, but not exclusively, for use in a thermosensitive stencil paper.

Thermosensitive stencil papers generally comprise a layer of a thin paper (usually having a basis weight of 5 to 15 gsm and a thickness of 10 to 50 microns) capable of absorbing ink bonded to a thermosensitive polymeric sheet in which the stencil design (which may for example be comprised text, drawings or the like) is formed by a thermal process (e.g. using a mask and a thermal head or flash lamp). To effect a printing operation, the stencil paper is located on a stencil printing machine and the thin paper is loaded with ink. During printing, the ink passes through the apertures (defining the stencil pattern) in the polymeric film whereby the design may be printed onto a substrate.

The thin paper layer must satisfy a number of criteria, and in particular it must:

- I) be capable of being securely bonded to the polymeric sheet such that no delamination occurs,
- II) have good mechanical strength so that the stencil paper may be used for producing multiple (e.g. at least 3,000) printed copies of the design on the stencil printing machine, and
- III) must have good dimensional stability so that those areas of the paper which are exposed through the apertures in the polymeric film (and which are not therefore supported in direct face to face contact with the film) do not expand or contract as a result of the absorption of ink into these areas.

EP-A-0 451 269 (Asahi) discloses a resin-treated thin sheet for a heat sensitive stencil printing paper. The thin sheet has a basis weight of 5 to 15 g/m<sup>2</sup> and a thickness of 10 to 50µm and comprises at least 10% by weight of polyester fibres of defined characteristics. The thin sheet further comprises a urethane resin or epoxy

resin which is present at crossing points and surfaces of filaments in an amount of at most  $3\text{g/m}^2$  of the thin sheet.

The process disclosed in EP-A-0 451 269 for producing the thin sheet comprises preparing a fibre dispersion which may contain additives (e.g. paper strength increasing additives), preparing paper from the fibrous dispersion, drying the paper and winding it onto a roll. Paper from the roll is then treated with the polyurethane resin.

JP-A-7040676 (Asahi) discloses the production of stencil papers by producing a furnish containing an epoxy modified polyamide polyamine resin (a wet strength agent), forming paper from the furnish, and treating the formed paper with an epoxy compound, an acrylic polymer containing active hydrogen atoms and a urethane polymer also containing active hydrogen atoms. The treated paper is then dried.

According to the first aspect of the present invention there is provided a paper comprised of cellulose fibres and optionally synthetic polymer fibres wherein the fibres of the paper are bonded together by a cured resin admixture comprising 0.05 gsm to 1 gsm of dry strength increasing additive, 0.1 gsm to 0.5 gsm of a cationic wet strength increasing agent, and 0.5 gsm to 3 gsm of an acrylate polymer, the admixture having been added and cured subsequent to the paper forming step in a single operation.

According to the second aspect of the present invention, there is provided a method of producing a paper which is comprised of cellulose fibres and optionally synthetic polymeric fibres wherein after the paper forming step the web is impregnated with an aqueous resin admixture comprised of a dry strength increasing additive, a non-ionic or cationic dispersion of acrylate polymer (e.g. a styrene acrylate dispersion) and a cationic wet strength increasing agent in amounts such that the paper comprises 0.05 gsm to 1.00 gsm of the dry strength increasing additive, 0.1 gsm to 0.5 gsm of the wet strength increasing agent, and 0.5 gsm to 3 gsm of acrylate polymer, and the resin admixture is subsequently cured.

The use (after the paper forming step) of a resin admixture which is comprised of a dry strength increasing additive, a non-ionic or cationic dispersion of an acrylate

polymer and a cationic wet strength increasing agent and which is subsequently cured has the advantage that all resin components (including the wet strength increasing agents) are applied in a single operation. This is rendered possible by the use of a non ionic dispersion of an acrylate polymer and has the advantage that (by virtue of the single resin treatment step) the number of operations which cause creasing or paper damage is minimised. A further advantage is that the various resin components are able to cure with each other as well as with sites on the cellulose fibres so as to produce a cured impregnant which gives the paper good mechanical and dimensional stability. The cured dry strength increasing additive and acrylate polymer bond the fibres together at their crossing points to give good wet and dry strength characteristics for the finished paper. Addition of the wet strength increasing additive at the paper forming step would reduce the number of sites on the cellulose available for curing with the dry strength increasing additive and polyurethane.

Papers produced by the invention are eminently suitable for use in stencil papers for which purpose the paper of the invention preferably has a basis weight of 5 to 15 gsm and a thickness of 10 to 50 microns. The paper of the invention can however be used for a variety of applications where dimensional stability and good pore size distribution are required, e.g. aqueous filtration. For such applications it is possible for the paper to have a basis weight above 15 gsm.

When used in stencil papers, the acrylate polymer of the thin paper provides a good lamination of the thermosensitive polymeric film (usually polyester) to the paper. For use in stencils, the papers of the invention may incorporate polymeric fibres as an aid to dimensional stability. Such fibres would generally be present in an amount of 10% to 50% by weight of the paper and may for example be polyester fibres. Suitable polyester fibres are as described in EP-A-0 451 269. The cured impregnant in the paper of the invention serves to "key" such polymeric fibres into the paper and prevent these fibres being "pulled out" either during lamination to the thermosensitive polymeric film or during actual printing.

The acrylate polymer used in the invention may be a homopolymer or copolymer. The polymer may for example be obtained by homo- or co-

polymerisation of a  $C_{1-4}$  ester of acrylic or methacrylic acid. Thus polymers for use in the invention may be obtained by homo- or co-polymerisation of methyl (meth)acrylate, ethyl (meth)acrylate, propyl (meth)acrylate or butyl (meth)acrylate. Particularly preferred copolymers for use in the invention are obtained by polymerisation of at least one (meth)acrylate monomer with a compound (other than a (meth)acrylate) having at least one olefinically unsaturated double which (in the case where two or more such bonds are present) may be conjugated or non-conjugated. A preferred example of such an olefinically unsaturated comonomer is styrene. A further example is butadiene.

Copolymers of the (meth)acrylate and olefinically unsaturated monomer may be random, block, or graft copolymers and preferably comprise 25-75% of the olefinically unsaturated monomer, 25-75% of (meth)acrylate and optionally up to 10% of a functional monomer, e.g. (meth)acrylic acid, acrylamide or N-methylol acrylamide.

A preferred acrylate copolymer for use in the invention is a styrene/meth(acrylate) copolymer.

The acrylate polymer is preferably provided as an aqueous non-ionic or cationic dispersion.

By "non-ionic" dispersion we mean that particles of the acrylate polymer are maintained in suspension in a continuous aqueous phase by a non-ionic surfactant. Similarly a "cationic" dispersion is one which employs a cationic surfactant to maintain the acrylate polymer in suspension.

The dispersion may comprise 40% to 80% by weight of the dispersed phase.

For preference the dispersion is a non-ionic dispersion.

Preferably the non-ionic dispersion of the acrylate copolymer has a pH of about 3.0, a viscosity of about 600 mPa.s at 25°C, and a specific gravity of about 1.06 g cm<sup>-3</sup> at 25°C.

A particularly suitable acrylate copolymer dispersion for use in the invention is available from Rohm & Haas under the name PRIMAL HA16.

The dry strength increasing additive for use in the invention is preferably either carboxymethyl cellulose or polyvinyl alcohol. The most preferred dry strength additive is sodium carboxymethyl cellulose, preferably a cold water soluble low viscosity carboxymethyl cellulose with a degree of substitution 0.6-0.95 and a viscosity of 200-500 mPa.s at a solution concentration of 4% at 25°C. For preference the sodium carboxymethyl cellulose product used is non-film forming. A particularly suitable product is available from Meta Serla Chemicals under the name Cekol 30G. This product as a 1% solution in water, has a pH of 6.0-8.5 and readily chemically crosslinks with the cationic wet strength additive (which is preferably epoxidised polyamide-polyamine resin) in the admixture, to give high wet and dry strength properties to the cured tissue paper.

A polyvinyl alcohol may also be used as the dry strength increasing additive for use in the invention.

It is preferred that the polyvinyl alcohol has a degree of substitution of at least 95% and more preferably at least about 98%. Most preferably the polyvinyl alcohol is fully hydrolysed (98-99% hydrolysis) or a superhydrolysed (99+% hydrolysis) polyvinyl alcohol solution. It is however within the scope of the present invention to use a cold water soluble grade of polyvinyl alcohol, for example one having a degree of hydrolysis of 66 to 68%. Most preferably the polyvinyl alcohol is of a relatively high molecular weight exhibiting a solution viscosity (at 25% solids content) of greater than 40 mPa.s, preferably 45-70 mPa.s.

A particularly suitable polyvinyl alcohol for use in the present invention is Airvol 165.

In certain circumstances, it may be necessary or desirable to ensure that the polyvinyl alcohol does not form a film and (in such circumstances) it is therefore necessary to use an agent for inhibiting film formation. This agent may for example be a non-ionic surfactant (e.g. Triton X114) used in an amount of 0.5-2% (e.g. about 1%) by weight of the polyvinyl alcohol present in the aqueous resin admixture.

Other dry strength increasing additives which may be used include amphoteric starch.

Preferred amphoteric starches for use in the invention have, at 25% concentration in water a pH of 5.0 to 7.0, a viscosity of 20-30 poise and a specific gravity of about  $1.15 \text{ g cm}^{-3}$ . A particularly suitable starch for use in the invention is SPP5400 liquid natural polymer available from National Starch Chemical. As supplied, SPP5400 comprises a 25% solution of the polymer. The solution is further diluted with water for use in the method of the invention.

The wet strength increasing agent used in the method of the invention is preferably a cationic epoxidised polyamide-polyamine resin or a cationic amino-formaldehyde resin, e.g. a melamine formaldehyde resin. A particularly suitable product is Kymene SLX2 available from Hercules. Other examples include Kenores from Akzo Nobel and BIP Melamine BC355.

As indicated, the aqueous resin admixture for use in the invention preferably comprises carboxymethyl cellulose or polyvinyl alcohol as the dry strength increasing additive. As such, the aqueous resin admixture is preferably formed by the following steps:

(a) dissolution of the carboxymethyl cellulose in water at (e.g. at  $10-30^{\circ}\text{C}$ ) to form a fully dissolved solution, or

dissolution of the polyvinyl alcohol in water followed by heating to  $95^{\circ}\text{C}$  to form a fully hydrolysed solution, followed by cooling to  $10-30^{\circ}\text{C}$  and addition of a surfactant;

(b) addition of the wet strength increasing agent to either of the dry strength increasing agent solutions obtained from (a); and

(c) addition of an aqueous non-ionic dispersion of the acrylate polymer resin to the admixture obtained from step (c).

The aqueous resin admixture may be impregnated into the preformed paper in a single pass at a coating station which could for example involve application of the impregnant by means of a size press or gravure roll.

The amount of aqueous resin admixture applied to the paper is such that the latter is treated with 0.05 gsm to 1 gsm of carboxymethyl cellulose or polyvinyl alcohol, 0.1 gsm to 0.5 gsm of the wet strength increasing agent, and 0.5 gsm to 3 gsm

of the acrylate polymer. Impregnation of carboxymethyl cellulose or polyvinyl alcohol in an amount greater than 1 gsm and/or impregnation of polyurethane or acrylate polymer in an amount greater than 3 gsm of the paper results in film formation of these resins across the voids of the paper resulting (when the paper is to be used in a stencil paper) in insufficient ink transmission, or poor ink fill-in, where the stencil master has poor optical density or poor definition of characters during printing. Use of more than 0.5 gsm of the wet strength increasing additive gives similar disadvantages. Use of amounts of the various resins below specified minima gives rise to problems such as unsatisfactory wet strength and unsatisfactory print durability.

The subsequent cross-linking by curing of the resin admixture may be effected in a single pass through a hot air dryer or an oven. Most preferably this curing step is effected at a temperature of 150 to 240°C. Curing temperatures below 150°C may result in insufficient bonding of polyvinyl alcohol and the acrylate copolymer at the crossing points on the fibre filaments leading to poor wet and dry strengths which in turn lead to poor print durability of the stencil master. A curing temperature above 240°C may result in decomposition of the polyvinyl alcohol and the acrylate copolymer leading to poor bonding at the fibre filament crossing points. This will give poor wet and dry strengths again leading to poor print durability.

Papers in accordance with the invention may be formed into stencil papers in accordance with standard procedures. The advantageous strength and dimensional stability properties of the papers of the invention produce stencils which produce good definition print for several thousand copies.

The paper of the present invention comprises cellulose fibres and optionally, synthetic polymer fibres. The cellulose fibres may be natural bast fibres, e.g. derived from *musa textilis*. For example, the fibres may be of abaca or Manila Hemp. Alternatively the fibres may be flax or sisal.

The invention is illustrated by the following non-limiting Examples and Comparative Example.



### Examples and Comparative Examples

A range of papers were prepared from previously alkali cooked and washed vegetable fibres, which after fibrillating to 18°SR freeness had a fibre length of 5mm and a diameter of 15 microns. The vegetable fibres were then combined with polyester fibres of length 5mm and diameter 8 microns to form papers.

The papers were formed with different basis weights and after the paper formation step were treated with resin impregnants comprising either carboxymethyl cellulose or polyvinyl alcohol, epoxidised polyamide-polyamine (provided by Kymene SLX2) and acrylate polymer dispersion (provided by Rhone & Haas Primal HA16). The resin impregnant was produced in accordance with steps (a)-(c) above. Various impregnants were used to give the amount of either carboxymethyl cellulose or polyvinylalcohol, epoxidised polyamide-polyamine and acrylate polymer on the finished paper as shown in Table 1 below.

After impregnation the resin was cured at 180°C. The physical properties of the papers thus obtained are shown in Table 1 together with an assessment of the properties of print optical density and durability of printing when using the papers in stencils based upon the wet MD tensile strength data.

Examples 1 and 2 comprised 85% abaca fibres and 15% polyester fibres, these percentages being based on the total weight of fibres.

Example 3 comprised 85% mixed vegetable fibres (providing 40% (of 85%) by weight abaca) and 15% polyester fibres, these percentages being based on total weight of fibres.

Example 4 comprised 85% mixed vegetable fibres (providing 80% (of 85%) abaca) and 15% polyester fibres, these percentages based on total weight of fibres.

Examples 5-7 comprised 85% abaca fibres and 15% polyester fibres.

Comparative Example 1 comprised 85% abaca fibres and 15% polyester fibres, these percentages being based on total weight of fibres.

The papers were formed with differing basis weights and after the paper formation step were treated with resin impregnants comprising either carboxymethyl cellulose or polyvinyl alcohol, epoxidised polyamide-polyamine (provided by

Kymene SLX2) and acrylate polymer dispersion (provided by Rohm & Haas PRIMAL HA16). The resin impregnant was produced in accordance with steps (a)-(c) above. Various impregnants were used to give the amount of either carboxymethyl cellulose or polyvinyl alcohol, epoxidised polyamide-polyamine and acrylate copolymer on the finished paper as shown in Table 1 below.

After impregnation the resin was cured at 180°C. The physical properties of the papers thus obtained are shown in Table 1 together with an assessment of the properties of print optical density and durability of printing when using the papers in stencils based upon the wet md tensile strength data.

TABLE I

RESIN IMPREGNATE	EX1	EX2	EX3	EX4	EX5	EX6	EX7	COMP EX1	COMP EX2	COMP EX3	COMP EX4	COMP EX5
POLYVINYL ALCOHOL (g/m <sup>2</sup> )	0.21	-	-	-	0.05	0.05	-	0.10	1.20	-	-	-
EPOXIDISED (g/m <sup>2</sup> ) POLYAMINE	0.25	0.10	0.10	0.10	0.50	0.14	0.23	0.15	0.60	0.02	0.00	0.10
ACRYLATE POLYMER (g/m <sup>2</sup> )	1.00	0.50	1.10	1.00	0.87	2.50	0.50	0.1	1.00	0.10	0.10	3.25
CARBOXYMETHYL CELLULOSE	-	0.28	0.28	0.28	-	-	0.80	-	-	0.28	1.20	0.28
PHYSICAL PROPERTIES BASIS WEIGHT (g/m <sup>2</sup> )	11.7	11.0	12.2	12.1	12.1	12.1	12.3	10.8	12.8	11.7	12.5	12.0
CALIPER (microns)	44	43	42	42	43	42	45	45	46	42	48	43
APPARENT DENSITY (g/cm <sup>3</sup> )	0.27	0.26	0.29	0.29	0.28	0.29	0.27	0.26	0.28	0.28	0.26	0.28
DRY TENSILE MD (g/mm)	80	64	72	72	73	88	90	47	82	55	66	95
WET TENSILE MD (g/mm)	38	29	39	40	33	43	28	14	45	16	12	44
AIR PERMEABILITY (m <sup>3</sup> /min/m <sup>2</sup> )	220	245	230	265	250	220	205	260	175	245	170	165
PRINTING CHARACTERISTICS												
PRINTING	GOOD	GOOD	GOOD	GOOD	GOOD	GOOD	GOOD	GOOD	POOR	GOOD	POOR	POOR
OPTICAL DENSITY												
PRINTING DURABILITY	GOOD	GOOD	GOOD	GOOD	GOOD	GOOD	GOOD	POOR	GOOD	POOR	POOR	GOOD

It will be seen from Table 1 that the products of Examples 1 to 7 in (i.e. products in accordance with the invention) had wet strength, printing optical density and print durability characteristics rendering them suitable for use in a thermosensitive stencil paper. On the other hand, the paper of Comparative Examples 1 to 5 did not have the required characteristics for use in a thermosensitive stencil paper.

**CLAIMS**

1. A paper comprised of cellulose fibres and optionally synthetic polymer fibres wherein the fibres of the paper are bonded together by a cured resin admixture comprising 0.05 gsm to 1 gsm of dry strength increasing additive, 0.1 gsm to 0.5 gsm of a cationic wet strength increasing agent, and 0.5 gsm to 3 gsm of an acrylate polymer, the admixture having been added and cured subsequent to the paper forming step in a single operation.
2. A paper as claimed in claim 1 wherein the acrylate copolymer is a styrene-acrylate copolymer.
3. A paper as claimed in claim 1 or 2 wherein the dry strength increasing additive is polyvinyl alcohol.
4. A paper as claimed in claim 1 to 3 wherein the dry strength increasing additive is carboxymethyl cellulose.
5. A paper as claimed in any one of claims 1 to 4 wherein the wet strength increasing agent is a cationic epoxidised polyamide-polyamine resin or a cationic amino-formaldehyde resin.
6. A paper as claimed in any one of claims 1 to 5 having a basis weight of 5 to 15 gsm and a thickness of 10 to 50 microns.
7. A paper as claimed in any one of claims 1 to 6 incorporating 10% to 50% by weight of the paper of synthetic polymeric fibres.
8. A paper as claimed in claim 7 wherein the synthetic polymeric fibres are polyester fibres or acrylic fibres.

9. A paper as claimed in any one of claims 1 to 8 wherein the cellulose fibres comprise bast natural fibres.
10. A paper as claimed in claim 9 wherein the bast natural fibres are manila hemp, flax, jute or sisal fibres.
11. A thermosensitive stencil paper comprising a paper as claimed in any one of claims 1 to 9 bonded to a thermosensitive polymeric sheet.
12. A method of producing a paper which is comprised of cellulose fibres and optionally synthetic polymeric fibres wherein after the paper forming step the web is impregnated with an aqueous resin admixture comprised of a dry strength increasing additive, a non-ionic or cationic dispersion of acrylate polymer and a cationic wet strength increasing agent in amounts such that the paper comprises 0.05 gsm to 1.00 gsm of the dry strength increasing additive, 0.1 gsm to 0.5 gsm of the wet strength increasing agent, and 0.5 gsm to 3 gsm of acrylate polymer, and the resin admixture is subsequently cured.
13. A method as claimed in claim 12 wherein the acrylate copolymer is a styrene-acrylate copolymer.
14. A method as claimed in claim 12 or 13 wherein the non-ionic dispersion of the acrylate copolymer has a pH of about 3.0, a viscosity of about 600 mPa.s at 25°C, and a specific gravity of about 1.06 g cm<sup>-3</sup> at 25°C.
15. A method as claimed in any one of claims 12 to 14 wherein the dry strength increasing additive is carboxymethyl cellulose.

16. A method as claimed in claim 15 wherein the carboxymethyl cellulose is cold water soluble, has a degree of substitution of 0.6 to 0.95 and a viscosity of 200 to 500 mPa.s at a solution concentration of 4% at 25°C.

17. A method as claimed in claim 15 or 16 wherein the aqueous resin admixture is formed by the following steps:

(a) dissolution of the carboxymethyl cellulose in water to form a fully dissolved solution;

(b) addition of the wet strength increasing agent to the solution obtained from (a); and

(c) addition of an aqueous non-ionic dispersion of the acrylate polymer resin to the admixture obtained from step (c).

18. A method as claimed in claims 12 to 14 wherein the dry strength increasing additive is polyvinyl alcohol and an agent is used to inhibit the film forming characteristics of the polyvinyl alcohol.

19. A method as claimed in claim 18 wherein the polyvinyl alcohol has a degree of hydrolysis of at least 95%.

20. A method as claimed in claim 19 wherein the polyvinyl alcohol has a degree of hydrolysis of at least 98%.

21. A method as claimed in claim 20 wherein the polyvinyl alcohol has a degree of hydrolysis of at least 99%.

22. A method as claimed in any one of claims 18 to 21 wherein the polyvinyl alcohol exhibits a solution viscosity at 4% solid content of greater than 40 cP.
23. A method as claimed in any one of claims 18 to 22 wherein the agent for inhibiting the film forming characteristics of the polyvinyl alcohol is a non-ionic surface active agent.
24. A method as claimed in any one of claims 18 to 23 wherein the aqueous resin admixture is formed by the following steps:
- a) dissolution of polyvinyl alcohol in water to form a fully hydrolysed solution;
  - b) addition of a surface active agent to inhibit film forming characteristics of polyvinyl alcohol;
  - c) addition of the wet strength increasing agent to the polyvinyl alcohol solution; and
  - d) addition of a non ionic aqueous dispersion of the polyurethane resin or ethylene vinyl acetate to the admixture obtained from step (c).
25. A method as claimed in any one of claims 12 to 24 wherein the wet strength increasing agent is a cationic epoxidised polyamide-polyamine resin, or a cationic malamine formaldehyde resin.
26. A method as claimed in any one of claims 12 to 25 wherein the resin is cured at a temperature of 150 to 200°C.





Application No: GB 9805452.1  
Claims searched: 1-26

Examiner: Carol Davies  
Date of search: 13 August 1997

**Patents Act 1977**  
**Search Report under Section 17**

**Databases searched:**

UK Patent Office collections, including GB, EP, WO & US patent specifications, in:

UK Cl (Ed.P): D1R (RDH); G2C (CHX)

Int Cl (Ed.6): B41N 1/24; D21H 17/34, 17/37

Other:

**Documents considered to be relevant:**

Category	Identity of document and relevant passage	Relevant to claims
A	EP 0451269 A1 (ASAHI) See Examples 1-8	
A	WPI Abstract Accession Number 95-118445/199516 & JP 7040676 A (ASAHI) 10.02.95 (see abstract)	

X	Document indicating lack of novelty or inventive step	A	Document indicating technological background and/or state of the art.
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